IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Michael SCHMIDT, et al.: Group Art Unit: 1626

Serial No.: 09/877,259 Examiner:

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For: IONIC LIQUIDS II

DECLARATION UNDER 37 C.F.R. § 1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

SIR:

I, Nikolai Ignatyev, am a citizen of the Ukraine residing at Duisburg, Germany;

I am a physical and organic chemist by training and experience.

The degree „Doctor of Science" (at least equivalent to PhD) was bestowed on me by the National Academy of Science, Institute of Organic Chemistry of Kiev, Ukraine, in 1980.
Second promotion to the position of Senior Researcher (at least equivalent to the position of Docent at the University) was bestowed on me by the National Academy of Science, Institute of Organic Chemistry of Kiev, Ukraine, in 1988.

Since 1 of June, 2000, I have been employed as a research scientist in the NBC-Department of Merck KGaA, Darmstadt, Germany.

I am author or co-author of numerous papers and patents in the fields of Organic Electrochemistry and Chemistry of Fluorine Compounds. I am a Member of the Electrochemical Society (Pennington, NJ 08534-2839 USA) since 1997.

The following comparative study of thermal stability of Ionic Liquids with $[(CF_3)_3PF_3]^-$ and $[(C_2F_5)_3PF_3]^-$ -anions was conducted under my supervision:

1. The samples of ionic liquids with $[(CF_3)_3PF_3]^-$ and $[(C_2F_5)_3PF_3]^-$ -anions were prepared by the reaction between 1-hexyl-3-methyl imidazolium chloride and potassium tris(trifluoromethyl)trifluorophosphate or potassium tris(pentafluoroethyl)trifluorophosphate (anion exchange reaction) in water. Ionic liquids with $[(CF_3)_3PF_3]^-$ and $[(C_2F_5)_3PF_3]^-$ -anions are not soluble in water and can be easily separated from water solution. After washing with water and drying in vacuum these ionic liquids were obtained as pure substances (the structures were confirmed by ^1H, ^{19}F and ^{31}P MNR spectra).

2. The thermal stability of 1-hexyl-3-methyl imidazolium ionic liquids with $[(CF_3)_3PF_3]^-$ and $[(C_2F_5)_3PF_3]^-$ -anions was studied by DSC-method. DSC-diagrams (pages 4 and 5 of the declaration) show that:
   1-Hexyl-3-methyl imidazolium $[(CF_3)_3PF_3]$ starts to decompose at 251°, intensive exothermic decomposition takes place at 293° C and the top of the exothermic peak is appeared at 327° C.
   1-Hexyl-3-methyl imidazolium $[(C_2F_5)_3PF_3]^-$ starts to decompose at 263° C, intensive exothermic decomposition takes place at 320° C and the top of the exothermic peak is appeared at 366° C.
   These data show the prior thermal stability of ionic liquid with $[(C_2F_5)_3PF_3]^-$ -anions.

3. Long time thermal stability of 1-hexyl-3-methyl imidazolium ionic liquids with $[(CF_3)_3PF_3]^-$ - and $[(C_2F_5)_3PF_3]^-$ -anions was studied in the following way: the both samples were placed into the glass tube closed with the rubber cork and were heated in metal-bath at 250° C for 7 hours. After this time the NMR spectra were measured for both samples. The spectra indicate (pages 6 to 9 of the declaration) that in the case of ionic liquid with $[(CF_3)_3PF_3]^-$ -anion, beside the reaction with glass, an intensive decomposition with the loosing of CF_3-groups takes place (substantial pressure was developed in the tube and the cork was knocked out at the end of the heating).

More than 50 % of starting material was destroyed at this temperature within 7 hours (spectra pages 8 and 9 of the declaration).

Opposite, the ionic liquid with $[(C_2F_5)_3PF_3]^-$ -anion shows much better thermal stability than the ionic liquid with $[(CF_3)_3PF_3]^-$ -anion. Beside slow reaction with the glass (less intensive than in the case of $[(CF_3)_3PF_3]^-$ -anion), no decomposition of ionic liquid with $[(C_2F_5)_3PF_3]^-$ -anion takes place at this temperature according to the MNR spectra (pages 10 to 13 of the declaration).

The spectra were recorded by Bruker ARX 400 Spectrometer. The frequency for the ^{31}P nucleus was: 161.98 MHz.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date:

Nikolai (Mykola) Ignatyev

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